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C7D

# (54) Process for obtaining metallic titanium from an anatase concentrate by an alumino-thermic or magnesium-thermic method

(57) A low cost method of obtaining metallic titanium from anatase concentrates having a $TiO_2$ content of 70.0% to 99.9% using aluminium or magnesium powder (or molten aluminium) as reducing agent in a proportion of 20% to 50% of the reaction charge, in any kind of kiln, under vacuum, or in a noble gas atmosphere, or in a normal atmosphere. Preferably, a reaction accelerating agent is present in the charge in the form of a chlorate or nitrate of an alkali metal. CaO may be present as a melting agent, if desired, together with $CaF_2$.

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SPECIFICATION

**Process for obtaining metallic titanium from an anatase concentrate by an alumino-thermic or magnesium-thermic method**

The present invention relates to a process for obtaining metallic titanium from anatase concentrates having a $TiO_2$ content of from 70.0% to 99.9%, using alumino-thermic or magnesium-thermic methods.

By using the classical methods of obtaining metallic titanium from concentrates of titanium ores, such as by the Kroll process, which involves the chlorination of the concentrate with a subsequent reduction of the tetra-chloride obtained by an alkali metal or by metallic magnesium in a noble gas atmosphere or under vacuum, a sponge is obtained, which is subsequently calcined. Another method of producing metallic titanium is the electrolysis in molten salt of titanium iodide.

The processes referred to above are expensive, thus raising the price of the final product, which prevents it from being more widely used in industry.

For this reason new research has been carried out, with the object of lowering the costs of producing metallic titanium, so that it can be used on a wider industrial scale.

Various tests were performed using alumino-thermic and magnesium-thermic processes until a highly satisfactory product was obtained. These tests led to the present invention

According to the invention, therefore, there is provided a process for obtaining metallic titanium from an anatase concentrate having a TiO content of from 70.0% to 99.9%, by an alumino-thermic or magnesium-thermic method using aluminium metal or magnesium metal as reducing agent as a proportion of 20% to 50% of the reaction charge,in a kiln of any type, under vacuum, or in a noble gas atmosphere, or in a normal atmosphere.

While not essential, it is preferred to include in the reaction charge, one or more reaction accelerating agents to furnish extra heat for the process, such agents being selected from alkali metal chlorates and nitrates, such as those of sodium, potassium and lithium.

A preferred reaction charge in the process of the invention is basically composed of:

a) an anatase concentrate having a $TiO_2$-content of from 70.0% to 99.9%;

b) one or more reaction accelerators to furnish extra heat for the reaction process, selected from the chlorates and nitrates of the following alkaline metals: sodium, potassium and lithium; and

c) as smelting material, calcium oxide pure, or together with fluorite.

The alumino-thermic or magnesium-thermic reaction to obtain metallic titanium from $TiO_2$ concentrates consists in forming a reaction-producing powder (particle size of 100% 100 mesh and 80% 325 mesh), whereby liquid metallic titanium, and an aluminium or magnesium slag are obtained. As explained hereafter, aluminium may also be used in molten form.

The particle size of the aluminium or magnesium powder, for better recuperation, corresponds to an average particle diameter of 130 μm.

Aluminium or magnesium powder, the titanium ore concentrate, and the oxidizing salt or salts (chlorates and alkaline nitrates) are mixed in adequate proportions. The mixture is homogenized and the materials must be in dry form.

The anatase concentrate is preferably present in the reaction charge in a proportion of 45% to 60% thereof. The reducing agent is preferably present in a proportion of 28% to 35% of the charge. The reaction accelerating agent(s) is preferably present in an amount of 6% to 13% of the reaction charge.

The reaction may be started by pre-heating, e.g. at a temperature of 350°C, or by resistance heating, or by the addition of an appropriate reagent (e.g. a barium salt, or the like). The addition of CaO to the mixture is an effective way of lowering the liquid temperature of the slag, but is not essential to the process. The CaO is preferably added in a proportion of 0.1% to 25% of the reaction charge. Similarly, the addition of fluorite ($CaF_2$) to the mixture is effective in liquifying the slags, but again not essential. When used, it is preferably added to the CaO melting agent in the proportion of 0.1% to 40% of the CaO. When using a kiln with a vacuum of over 1.0 torr (133.3 Pa), the alumino-thermic or magnesium-thermic process may be carried out by placing the mixture in the interior of the kiln, establishing a vacuum of over 1.0 torr (133.3 Pa), and effecting firing by electrical resistance heating.

For low cost production, molten aluminium may be used as the reducing agent, instead of aluminium powder. In using a vacuum kiln, the mixture of titanium concentrate, alkaline chlorate and molten aluminium is placed in the interior of the kiln, this being heated by induction at a temperature higher than the melting point of aluminium, so that the exothermic reaction may get started.

The melting of the aluminium may also be accomplished from outside the kiln.

The invention is illustrated by the following non-limitative examples:-

*Example 1*

| | |
|---|---|
| Reaction charge: - anatase concentrate with 75% of $TiO_2$ | - 52.64% |
| aluminium powder | - 31.57% |
| $NaClO_3$ | - 10.52% |
| CaO | - 5.26% |

Total weight of the reaction charge - 15.2 kg.

Slag formation occurs in well defined phases. The lower part has a high degree of metallization, showing the "geometric aspect" of the desired baloon.

Yield - 80% by weight of metallic titanium.

Open kiln.

*Example 2*

| | |
|---|---|
| Reaction charge: - anatase concentrate with 86.0% of $TiO_2$ | - 55.56% |
| aluminium powder | - 33.33% |
| $NaClO_3$ | - 11.11% |

Total weight of the charge - 14.4 kg.

Slag and metal formation in well defined phases with good separation.

Yield - 70% by weight of metallic titanium.

Open kiln.

*Example 3*

| | |
|---|---|
| Reaction charge: - anatase concentrate with 85.0% of $TiO_2$ | - 53.48% |
| aluminium powder | - 32.08% |
| $KClO_3$ | - 8.02% |
| CaO | 5.34% |
| $CaF_2$ | - 1.08% |

Total weight of the reaction charge - 18.7 kg.

Slag formation in well defined phases with better fluidity and excellent separation of the phases.

Yield - 90.0% by weight of metallic titanium.

Closed vacuum kiln.

*Example 4*

| | |
|---|---|
| Reaction charge: - anatase concentrate with 94.0% of $TiO_2$ | - 48.78 |
| aluminium powder | - 32.52% |
| $NaClO_3$ | - 12.19% |
| CaO | - 4.06% |
| $CaF_2$ | - 2.45% |

Total weight of the charge - 24.6 kg.

Slag and metal formation in well defined phases, good fluidity of the slags and excellent phase separation.

Yield - 93.0% of metallic titanium.

Vacuum closed kiln.

In the Specification, all percentages are by weight.

CLAIMS

1. A process for obtaining metallic titanium from an anatase concentrate having a $TiO_2$ content of from 70.0% to 99.9% by an alumino-thermic or magnesium- thermic method using aluminium metal or magnesium metal as reducing agent as a proportion of 20% to 50% of the reaction charge in a kiln of any type, under vacuum, or in a noble gas atmosphere, or in a normal atmosphere.

2. A process as claimed in Claim 1, wherein the anatase concentrate is present in the reaction charge as a proportion of 45.0% to 60.0% of the total charge.

3. A process as claimed in Claim 1 or Claim 2, wherein the aluminium or magnesium reducing agent in powder form is present in the reaction charge in the proportion of 28% to 35% of the charge.

4. A process as claimed in any one of Claims 1 to 3, wherein at least one reaction accelerating agent which furnishes extra heat for the reaction is present in the reaction charge.

5. A process as claimed in Claim 4, wherein said reaction accelerating agent is a chlorate or nitrate of an alkali metal.

6. A process as claimed in Claim 4 or Claim 5, wherein said reaction accelerating agent or agents is or are present in the proportion of 6% to 13% of the reaction charge.

7. A process as claimed in any one of the preceding Claims, wherein calcium oxide is present as a melting agent in the reaction charge in the proportion of 0.1% to 25% of the reaction charge.

8. A process as claimed in Claim 7, wherein fluorite ($CaF_2$) is added to the calcium oxide melting agent in the proportion of 0.1% to 40% of the amount of melting agent.

9. A process for obtaining metallic titanium from an anatase concentrate substantially as hereinbefore described with reference to any of the Examples.

10. Metallic titanium obtained by a process as claimed in any one of Claims 1 to 9.

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